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ATTORNEY'S DOCKET NUMBER

**TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371**

025219-268

U.S. APPLICATION NO. (If known, see 37 C.F.R. 1.5)

Unassigned

09/600590

INTERNATIONAL APPLICATION NO.
PCT/FR99/00187INTERNATIONAL FILING DATE
29, January, 1999 (29.01.99)PRIORITY DATE CLAIMED
30, January, 1998 (30.01.98)TITLE OF INVENTION
COMPLIANT SUBSTRATE IN PARTICULAR FOR HETERO-EPITAXIAL DEPOSITINGAPPLICANT(S) FOR DO/EO/US
Bernard ASPAR, Michel BRUEL, Eric JALAGUIER and Hubert MORICEAU

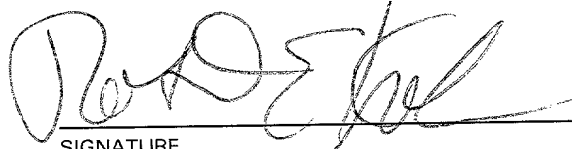
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and the PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US)
- ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
- ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made.
- ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☒ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.
☐ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information:

PCT Request, International Search Report and cited references

U.S. APPLICATION NO. (If known, see 37 C.F.R. 1.50) Unassigned 09/600590		INTERNATIONAL APPLICATION NO. PCT/FR99/00187		ATTORNEY'S DOCKET NUMBER 025219-268	
17. <input checked="" type="checkbox"/> The following fees are submitted:				CALCULATIONS	PTO USE ONLY
Basic National Fee (37 CFR 1.492(a)(1)-(5)): Search Report has been prepared by the EPO or JPO \$840.00 (970) International preliminary examination fee paid to USPTO (37 CFR 1.482) \$670.00 (956) No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)) \$690.00 (958) Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$970.00 (960) International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4) \$96.00 (962)					
ENTER APPROPRIATE BASIC FEE AMOUNT =				\$ 840.00	
Surcharge of \$130.00 (154) for furnishing the oath or declaration later than months from the earliest claimed priority date (37 CFR 1.492(e)). 20 <input type="checkbox"/> 30 <input type="checkbox"/>				\$ 0.00	
Claims	Number Filed	Number Extra	Rate		
Total Claims	34 -20 =	14	X\$18.00 (966)	\$ 252.00	
Independent Claims	1 -3 =	0	X\$78.00 (964)	\$ 0.00	
			+\$260.00 (968)	\$ 0.00	
Multiple dependent claim(s) (if applicable)					
TOTAL OF ABOVE CALCULATIONS =				\$ 1,092.00	
Reduction for 1/2 for filing by small entity, if applicable. Verified Small Entity statement must also be filed. (Note 37 CFR 1.9, 1.27, 1.28).				\$ 0.00	
SUBTOTAL =				\$ 1,092.00	
Processing fee of \$130.00 (156) for furnishing the English translation later than months from the earliest claimed priority date (37 CFR 1.492(f)). 20 <input type="checkbox"/> 30 <input type="checkbox"/>				\$ 0.00	
TOTAL NATIONAL FEE =				\$ 1,092.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). per property +				\$ 40.00	
TOTAL FEES ENCLOSED =				\$ 1,132.00	
				Amount to be:	
				refunded	\$
				charged	\$
a. <input checked="" type="checkbox"/> A check in the amount of \$ <u>1,132.00</u> to cover the above fees is enclosed. b. <input type="checkbox"/> Please charge my Deposit Account No. <u>02-4800</u> in the amount of \$_____ to cover the above fees. A duplicate copy of this sheet is enclosed. c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>02-4800</u> . A duplicate copy of this sheet is enclosed.					
NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.					
SEND ALL CORRESPONDENCE TO: Robert E. Krebs, Esq. BURNS, DOANE, SWECKER & MATHIS, L.L.P. P.O. Box 1404 Alexandria, Virginia 22313-1404					
 SIGNATURE					
Robert E. Krebs NAME					
<u>25,885</u> REGISTRATION NUMBER					

Patent
Attorney's Docket No. 025219-268

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of)	
Bernard ASPAR et al.)	Group Art Unit: Unassigned
Application No.: Unassigned)	Examiner: Unassigned
Filed: Herewith)	
For: COMPLIANT SUBSTRATE IN)	
PARTICULAR FOR HETERO-)	
EPITAXIAL DEPOSITING)	

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

Prior to examination, please amend the subject application as follows:

IN THE CLAIMS:

- Claim 4, line 26, delete "either of claims 2 or 3" and insert -- Claim 2 --.
- Claim 7, line 5, delete "any of claims 2 to 6" and insert -- Claim 2 --.
- Claim 10, line 21, delete "any of claims 2 to 6" and insert -- Claim 2 --.
- Claim 12, line 32, delete "either of claims 10 or 11" and insert -- Claim 10 --.
- Claim 15, line 13, delete "either of claims 13 or 14" and insert -- Claim 13 --.
- Claim 18, line 27, delete "any of claims 1 to 17" and insert -- Claim 1 --.
- Claim 19, line 2, delete "any of the preceding claims" and insert -- Claim 1 --.
- Claim 22, line 19, delete "either of claims 20 or 21" and insert -- Claim 20 --.
- Claim 23, line 24, delete "any of claims 19 to 22" and insert -- Claim 19 --.

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Claim 24, line 28, delete "any of claims 19 to 23" and insert -- Claim 19 --.

PLEASE ADD THE FOLLOWING CLAIMS:

25. Process according to Claim 3, characterized in that doping agents are associated with the one or more gas species.
26. Process according to Claim 6, characterized in that said implantation is made via the substrate surface, the region lying between the substrate surface and the layer of microcavities providing said thin layer.
27. Process according to Claim 6, characterized in that said implantation is made via the substrate surface, this surface carrying a first thin layer, the region lying between the substrate and the layer of microcavities providing a second thin layer.
28. Process according to Claim 11, characterized in that implantation by bombardment is made via a sacrificial layer carried by the first thin layer, said sacrificial layer then being removed.
29. Compliant substrate according to Claim 14, characterized in that said joining zone also comprises at least one intermediate layer (22; 32, 33) between the thin layer (23; 34) and the carrier (21; 31).
30. Compliant substrate according to Claim 17, characterized in that the joining means comprise a layer of microcavities and a bonding interface arranged either above or below the layer of microcavities.

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31. Compliant substrate (5, 20, 30) according to Claim 18, characterized in that said thin layer (4, 13, 23, 34) is in a first crystalline material and is intended to be used as hetero-epitaxial growth seed for a second crystalline material forming said structure.
32. Compliant substrate according to Claim 21, characterized in that said foreign element is a doping agent of the thin layer.
33. Compliant substrate (5, 20, 30) according to Claim 22, characterized in that said first crystalline material is a semiconductor.
34. Application of the compliant substrate (5, 20, 30) according to Claim 23, to the hetero-epitaxial growth of a crystalline material chosen from among GaN, SiGe, AlN, InN and SiC.


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REMARKS

The claims of the subject application have been amended to avoid multiple dependency. Favorable consideration of the application is respectfully requested.

Respectfully submitted,

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Date: July 17, 2000

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COMPLIANT SUBSTRATE
IN PARTICULAR FOR HETERO-EPITAXIAL DEPOSITING

Technical field

This invention relates to a compliant substrate, that is to say a substrate able to accept stresses induced by a structure adhering to it, and which may be a layer deposited on a surface of this substrate by
5 hetero-epitaxy such that this layer suffers the least possible stress. It also relates to processes for obtaining such substrates.

Prior art

10 Electronic and optoelectronic applications demand a growing number of semiconductor materials and in particular compound semiconductors such as, for example, those of III-V type. However, at the present time it is only known how to fabricate solid substrates
15 for certain semiconductors such as silicon, gallium arsenide, silicon carbide and indium phosphide for example. For other semiconductors, the solution chosen is hetero-epitaxial growth on a substrate whose crystalline network is adapted to that of the
20 semiconductor layer which is to be grown.

However, this constraint of having to adapt lattice parameters at the interface of growth between layer and substrate severely limits the number and diversity of layers which may be grown, as it is only
25 rarely possible to find a substrate whose network is adapted to the desired layer. Hence, for example, no solid substrates exist which are perfectly adapted to the hetero-epitaxial growth of GaN, AlN and InN.

The use of ill-adapted substrates leads to the growth of layers of very poor quality. In particular, as soon as the thickness of the layer exceeds a critical value, which decreases the more the networks
5 are ill-adapted, the stresses are released in the hetero-epitaxial layer through the creation of structure defects (dislocations in particular).

To overcome these problems, specialists in epitaxy on thick substrates have used growth techniques which
10 include the fabrication of a stack of buffer layers whose purpose is to allow absorption of the stresses induced by the differences in lattice parameter between the substrate and the epitaxied layer, chiefly in the plane of epitaxy, and by the differences in thermal
15 dilatation coefficients between the two materials. In this latter case, the temperature at which the epitaxied layer is grown is also a parameter which has to be taken into account. This stack of buffer layers ends in a superficial layer which is therefore used to
20 germinate the epitaxied layer which is the desired layer. However, even using all this know-how, the materials obtained always contain crystalline defects and are frequently of insufficient quality to fabricate optoelectronic and/or electronic devices.

To remedy this problem, various studies on
25 compliant substrates have been conducted. By way of example, mention may be made of the article "New Approach to Grow Pseudomorphic Structures over the Critical Thickness" by Y.H. LO, published in the
30 journal Appl. Phys. Lett. 59 (18), 28 October 1991. In this area, the compliant substrate is in essence a crystalline substrate whose crystalline lattice (lattice parameter) is not necessarily adapted to the

layer it is desired to grow, but which, when the hetero-epitaxial layer is grown, has the property of relaxing the stresses related to growth of the layer, in the compliant substrate itself or at the interface, instead of allowing the stresses to relax in the hetero-epitaxial layer. In this way hetero-epitaxial layers of very high quality are obtained and, in principle, the compliant substrate allows growth of any type of layer on a crystalline network.

10 The fabrication of compliant substrates can be classified in three groups.

One first group relates to a very fine substrate (a few nm) that is self-supporting, which is very difficult to produce and even virtually impossible if it is required to obtain large surface areas. In this respect, reference may be made to the article: "Lattice Engineered Compliant Substrate for defect-free Hetero-epitaxial Growth" by F.E. EJECKAM et al., published in the journal Appl. Phys. Lett. 70 (13), 31 March 1997.

20 A second group relates to a SOI structure (Silicon-On-Insulator) on a substrate. In this case, the superficial film obtained is very thin and the underlying insulator layer is likely to undergo deformation under the effect of the temperature during growth of the thin film.

A third group relates to a structure of a so-called "twist bonding" type. In this case, the thin film, allowing stress relaxation, subsequently called compliance, is made by means of bonding, through molecular adhesion, two crystalline substrates of same type, whose crystalline networks are disoriented, and by thinning one of the substrates until only a very thin layer subsists. In this respect, reference may be

made to the article: "Dislocation-free InSb Grown on GaAs Compliant Universal Substrates" by F.E. EJECKAM et al., published in the journal Appl. Phys. Lett. 71 (6), 11 August 1997. This bonding with disorientation
5 induces, in the vicinity of the interface, the formation of dislocations which are found in the thinned layer, making the latter able to accommodate the stresses when a hetero-epitaxial layer is grown above it.

10 These compliant substrates of the prior art have certain limitations in use. For the self-supporting film, the limitation resides in the difficulty or virtual impossibility to produce a film of a few nm on a surface of several mm², and even more so, several
15 dozen cm². No material exists at these thicknesses that is sufficiently rigid for handling. For the SOI structure, the limitation resides in the imperfect compliance of the substrate. This is related to the capacity of the insulator to deform (even creep) in
20 order to absorb stresses. To achieve this result, recourse must be made to heat treatments at high temperatures and/or to adapted compositions (for example B and P doping for insulators of SiO₂ type). These heat treatments are not always compatible with
25 the layer to be epitaxied. For the third group of substrates, the difficulty is to obtain defect-free bonding over a large surface and to thin the layer down to a very narrow thickness. Also, this technique requires very good control over the crystalline
30 disorientation between the two substrates if it is desired to properly control the number and type of dislocations which impart the compliant nature to this type of structure.

It is also known that an intimate bond between two materials may be obtained by molecular adhesion. Several cases may be encountered in relation to the endings present on the surface at the time of bonding.

5 For example the terms hydrophilic or hydrophobic bonding are used.

Surface hydrophilia is generally obtained by means of chemical cleaning whose objective is to saturate the surface in OH hydroxyl groups (for silicon, for
10 example, a surface density of $4.6/\text{nm}^2$). Water molecules can then adsorb naturally on these sites. The contacting of the two surfaces thus prepared leads to their adhesion with significant bonding energy (0.1 J/m^2 for silicon oxide/silicon oxide bonding) even at
15 room temperature. Subsequent heat treatments allow its reinforcement owing to the development of the bonds present at the interface. Therefore, for $\text{SiO}_2\text{-SiO}_2$ bonding, heat treatments at low temperature, typically less than 300°C , bring the two surfaces together via
20 hydrogen bonds between vis-à-vis hydroxyl groups via the onset of initial Si-O-Si bonds. Bonding energy therefore increases regularly with temperature to reach a bonding energy of 2 J/m^2 at 900°C .

On the contrary, for hydrophobic bonding (that is
25 to say bonding which does not involve water molecules or hydroxyl groups), the surfaces are generally stripped before bonding in order to remove any native oxide. The cleaning used for stripping leaves the surfaces mainly saturated in Si-H endings, for silicon
30 for example. Bonding resistance is only assured by an attraction of Van der Waals type and the bonding energies measured at room temperature for silicon-silicon bonding (approximately 10 mJ/m^2) well relate to

the theoretical calculation. With temperature rise, Si-Si bonds are formed by reconstruction of the two contacted surfaces.

5 This bonding mechanism may occur for the majority of materials provided that their roughness and planarity are sufficiently low. These two methods used well demonstrate that it is possible to control bonding forces between the different contacted materials in relation to surface treatment, applied heat treatments
10 and surface roughness. One example of the development of this bonding energy is given in the article: "Mechanism for Silicon Direct Bonding" by Y. BACKLUND et al., published in the journal J. Micromech Microeng. 2 (1992), pages 158-160 (see figure 1 in particular).
15 This bonding energy is determined by a method which uses the propagation of a crack at the bonding interface under the effect of the insertion of a blade at the bonding interface and parallel to this interface.

20 As early as 1989, some authors mentioned the possibility of using molecular adhesion to produce bonding between a multilayer film of GaAs/InGaAs/GaAs, previously made on a substrate well adapted to this structure, and an oxidized silicon carrier. Specific
25 surface preparation enables low bonding forces to be obtained. In this respect, reference may be made to the article: "Characterization of Thin AlGaAs/InGaAs/GaAs Quantum-well Structures Bonded Directly to SiO₂/Si and Glass Substrates" by J.F. KLEM et al., published in the
30 journal J. Appl. Phys. 66 (1), July 1st 1989.

It is also known, for example through document FR-A-2 681 472, that implantation by bombardment of a rare gas or hydrogen in a semiconductor material, or in a

solid material whether crystalline or not (cf. FR-A-2 748 850) is able to create microcavities or platelets at a depth close to the average depth of penetration of the implanted species. The morphology (size, shape...) of these defects may change during heat treatments, in particular these cavities may have their size increased. Depending upon the type of material and especially depending upon its mechanical properties, these cavities may, according to the conditions of heat treatment, induce surface deformations called "blisters". The most important parameters that need to be controlled in order to obtain such deforming are the dose of gas inserted during implantation, the depth at which the gas species are implanted and the heat schedule applied during implantation. By way of example, an implantation of hydrogen in a silicon wafer at a dose of $3 \cdot 10^{16} \text{ H}^+/\text{cm}^2$, for an energy of 40 keV, creates a continuous embedded layer of microcavities that is approximately 150 nm thick, at an average depth of 330 nm. By continuous layer is meant a layer containing microcavities distributed in homogeneous manner over a certain thickness. These microcavities are of elongated shape (hence the name "platelets"). Their size is for example in the order of 6 nm in length and two atomic planes in thickness. If heat treatment is applied at 700°C for 30 minutes, the microcavities magnify and their size may increase for example from 6 nm to over 50 nm in length and by a few atomic planes at 4-6 nm in thickness. On the other hand, no disturbance of the implanted surface is noted. Cavity size and the pressure within these cavities are not sufficient to induce surface deformation. This provides a continuous layer of embedded defects with a

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zone containing microcracks (or microcavities or platelets) but with no surface deterioration.

The presence of microcavities is also seen in the case of implantation made by helium bombardment at the average depth of implantation R_p in a substrate, for example in silicon. In this case, the cavities obtained are present even at annealing temperatures in the order of 1000°C . These defects cause strong, deep weaknesses in the material.

Description of the disclosure

In order to remedy the disadvantages of the prior art, the present invention puts forward a compliant substrate which offers a thin layer of a material intended to be used to germinate hetero-epitaxial growth of another material. This thin layer is joined to the remainder of the substrate by joining means, which may be termed an embedded region, such that the thin layer and/or joining means accommodate all or part of the stresses caused during epitaxial growth of the epitaxied material, thereby preventing the occurrence of these stresses in the epitaxied material.

The compliant character of such a structure vis-à-vis a subsequently deposited material lies in the consideration given to differences in lattice parameter, thermal dilatation coefficients and the presence of the embedded region. By definition, the purpose of this compliant structure is to accommodate the stresses of the film of deposited material by relaxation thereof in the embedded region but possibly also in the thin layer.

One variant of the process consists of inserting a foreign element in the superficial thin film in order

to modify the crystallographic parameters of the thin layer forming the germination film for epitaxy and consequently to change its stress state before epitaxial growth of the layer to be obtained.

5 It has also been found that such a compliant substrate may in its principle be used to absorb stresses due to causes other than growth of a material by epitaxy. In fact this compliant substrate may be used to receive any stress-giving structure.

10 The purpose of the invention is therefore a compliant substrate comprising a carrier and at least one thin layer formed on the surface of said carrier and intended to receive, in integral manner, a stress-giving structure, the carrier and the thin layer being
15 joined one to another by joining means such that the stresses brought by said structure are absorbed in whole or in part by the thin layer and/or by the joining means, characterized in that said joining means comprise at least one joining zone chosen from among
20 the following joining zones: a layer of microcavities and/or a bonding interface whose bonding energy is controlled to permit the absorption of said stresses.

 The joining zone may be a layer of defects, for example a layer of microcavities. The layer of defects
25 may be created by implantation through bombardment of one or more gas species. These gas species may be chosen from among rare gases, hydrogen and fluorine. Doping agents may be associated with the one or more gas species. It is also possible to conduct diffusion
30 of the one or more implanted gas species. Implantation may be followed by heat treatment to enable the defects to develop. Implantation by bombardment may in particular be made via the substrate surface, the

region lying between the substrate surface and the layer of defects providing said thin layer. Optionally, the region lying between the substrate surface and the layer of defects is thinned to form said thin layer.

- 5 Implantation by bombardment may also be made through a sacrificial layer carried by said substrate surface, which said sacrificial layer can then be removed.

Implantation may be made via the substrate surface, this surface carrying a first thin layer, the
10 region between the substrate surface and the layer of microcavities providing a second thin layer. The layer of microcavities may be made in the vicinity of the interface between the first thin layer and the substrate. Implantation by bombardment may be made via
15 a sacrificial layer carried by the first thin layer, said sacrificial layer then being removed.

Bonding energy may be controlled by surface preparation and/or by heat treatment and/or through the creation of defects at this interface. These defects
20 may, for example, be created through implantation by bombardment and/or by bonding defects. This creation of defects generally allows weakening of the bonding interface. Surface preparation may be control of roughness and/or of hydrophilia. Wafer roughness may be
25 obtained by chemical attack with HF for example. Hydrophilia may be obtained by chemical cleaning of RCA type. The joining zone may also comprise at least one intermediate layer between the thin layer and the carrier. The intermediate layer may be made such that
30 it is formed of non-homogeneities able to relax the stresses. By way of example, mention may be made of grain joints, growth lines, inclusions, etc. This layer may be etched on all or part of its surface. The

intermediate layer may be a metal layer or a layer of a metal alloy.

The joining means may comprise a layer of microcavities and a bonding interface arranged either
5 above or below the layer of microcavities.

In one privileged application, the thin layer is in a first crystalline material and is intended to serve as a seed for hetero-epitaxial growth of a second crystalline material forming said structure. This thin
10 layer may be a layer that is pre-stressed through the insertion of a foreign element into said first crystalline material in order to promote the compliance of said substrate. The foreign body may be inserted through implantation by bombardment and/or inserted by
15 diffusion. This implantation may be made via a sacrificial oxide. This foreign element may be a doping agent of the thin layer. The first crystalline material may in particular be a semiconductor, for example Si or GaAs. Such compliant substrate may advantageously be
20 used for the hetero-epitaxial growth of a crystalline material chosen from among GaN, SiGe, AlN, InN and SiC.

Brief description of the figures

The invention will be better understood and other
25 advantages and special aspects will become apparent on reading the following description which is non-restrictive, accompanied by the appended drawings in which

- figures 1A to 1C illustrate a first example of
30 embodiment of a compliant substrate of the present invention, the joining zone being a layer of microcavities;

- figures 2A to 2C illustrate a second example of embodiment of a compliant substrate of the present invention, the joining zone comprising a bonding interface;

5 - figure 3 shows a compliant substrate of the present invention the joining zone comprising a bonding interface and an intermediate layer;

10 - figure 4 shows a compliant substrate of the present invention, the joining zone comprising a bonding interface between two intermediate layers;

 - figure 5 is a diagram illustrating the development of bonding energy for $\text{SiO}_2\text{-SiO}_2$ bonding in relation to temperature and surface roughness.

15 **Detailed description of embodiments of the invention**

 By way of preferred example, the remainder of the description shall relate to the fabrication of compliant substrates for the depositing of materials by hetero-epitaxy.

20 It is possible to obtain a film of narrow thickness from a substrate in which implantation of species is made (of ions for example) able to create, at a depth close to the average penetration depth of the species, a layer of defects which, between the
25 substrate surface and itself, delimits a film of narrow thickness. The species are chosen such that the layer of created defects is able to accommodate the stresses to which the film of narrow thickness may be subjected. The role of the layer of defects is also to carry the
30 film of narrow thickness (vertical action, perpendicular to the surface) while leaving it free of stresses in the horizontal plane (parallel to the surface). It may be necessary, in some cases, to apply

he treatment to the substrate after the implantation
 st as for example to increase defect size, to
 ca be defects to coalesce in clusters of greater
 s to modify their distribution to make the layer
 5 m to be adapted to stress accommodation.
 referably species are chosen from among rare
 ga hydrogen, or a combination of both, which are
 kn permit the creation of defects of microcavity
 ty, this case, a sufficient dose must be chosen to
 10 cre ese microcavities but lower than the critical
 dos e which implantation of species is likely to
 inc rface deformation of "blister" type. By way of
 exa in the case of silicon, it may be chosen to
 imp ydrogen ions at a dose of $3 \cdot 10^{16}/\text{cm}^2$. However,
 15 it e specified that this critical dose is related
 to tation conditions and type of doping.
 lm thickness is determined by the choice of
 imp ion energy. In order to produce a very thin
 fil ch is necessary to assure good compliance), a
 20 low ntation energy must be chosen. For example, in
 the of silicon and hydrogen ions, an energy will
 pre y be chosen in the range of 1 keV to 10 keV, a
 rar ich enables films to be produced having a
 thi of between 5 nm and 60 nm. It is also
 25 pos to obtain the required film thickness by
 thi (polishing, chemical attack, sacrificial
 ox:) a film obtained by implantation using energy
 tha greater than that which would have directly
 pro the required thickness.
 30 some cases, it may be advantageous to implant
 via rificial layer, for example a layer of silicon
 ox: n this case, it may no longer be necessary to
 use low energies. The removal of the sacrificial

layer may be sufficient to achieve a very thin superficial layer.

Figures 1A to 1C illustrate this last example. Figure 1A, from side view, represents a substrate 1, in
5 monocrystalline silicon for example, coated with a layer of silicon oxide 2 acting as sacrificial layer. Figure 1B represents an ion implantation step, with hydrogen ions, of substrate 1 via oxide layer 2. Implantation is made under the conditions determined
10 above. A layer 3 of microcavities or platelets is obtained determining a layer or thin film 4 adjacent to the oxide layer 2. On account of the presence of this oxide layer, the thickness of the thin layer 4 may be reduced and very precisely adjusted. The oxide layer 2
15 is then removed by chemical attack and compliant substrate 5 is obtained shown in figure 1C in which the assembly formed by the layer of microcavities 3 and the thin layer 4 (used as seed for a material to be epitaxied) forms a compliant layer. Optionally, heat
20 treatment may be applied to increase the size of the microcavities of layer 3.

For some applications, ion implantation may also be made via two monocrystalline layers. A first monocrystalline layer, formed in the substrate itself,
25 has a thickness between the substrate surface and the layer of microcavities induced by implantation. A second monocrystalline layer may be deposited on or transferred to the substrate. By way of example, the substrate chosen may be a structure made up of a thin
30 layer of GaAs (for example 3 nm thick) transferred onto a silicon substrate using a method such as that described in document FR-A-2 681 472 associated with thinning by means of sacrificial layers. Subsequently,

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a sacrificial layer of silicon oxide is deposited on the structure in order to allow hydrogen implantation at the required depth. Hydrogen implantation in the silicon is made by crossing through the sacrificial oxide layer and the GaAs layer to create microcavities in the silicon but at a depth very close to that of the GaAs/Si interface, for example at a depth in the order of a few nm, even a few dozen nm. One variant of embodiment may consist of creating microcavities in the vicinity of the interface between GaAs and silicon.

As indicated previously, the bonding forces are dependent upon numerous parameters (type of chemical species on the surface, heat schedule provided, initial surface roughness). However, these forces may be controlled so as to be able to control bonding energy. These bonding forces may then be accommodated in relation to the stress caused by the presence of a thin epitaxied layer of a material and induced by differences in lattice parameter, thermal dilatation coefficient, but also giving consideration to stresses induced by bonding through molecular adhesion itself. By way of example, for hydrophilic bonding of monocrystalline silicon wafers and using a method of fabricating a thin film of semiconductor material such as the one described in document Fr-A-2 681 472, it is possible to obtain a very thin layer of silicon (less than 10 nm) on an oxide layer of very narrow thickness (less than 5 nm). The originality in this case, compared with the process disclosed in document FR-A-2 681 472, lies in the final control of bonding forces, that is to say after fracture by annealing at low temperature (typically 450°C for 30 minutes for a hydrogen implantation dose in the order of $6.10^{16} \text{H}^+/\text{cm}^2$)

and mechanical-chemical polishing. One example of bonding forces which may be obtained is shown in the graphs in figure 5. For example, for $\text{SiO}_2\text{-SiO}_2$ bonding with a surface roughness of 6.25 Å rms (AFM measurement on 1 x 1 µm analysed surfaces) for the two contacted surfaces, bonding energies in the order of 250 mJ/m² are obtained even after treatment at 800°C.

Figures 2A to 2C illustrate this example of embodiment. Figure 2A shows, from side view, a substrate 10 in monocrystalline silicon of which one surface is coated with a very fine layer of silicon oxide 11. Via the oxide layer 11, hydrogen ions are implanted intended to induce a fracture zone. A layer of microcavities 12 is obtained determining, between itself and oxide layer 11, a very thin region 13 of silicon. Figure 2B shows, also from side view, another silicon substrate 14 coated with a very thin layer of silicon oxide 15. Substrates 10 and 14 are made integral by molecular adhesion of their oxide layers 11 and 15. Subsequently, through appropriate heat treatment, the microcavities of layer 12 are caused to coalesce to obtain fracture and separation of substrate 10 into two parts. The free surface of region 13 is polished to form a thin layer intended for hetero-epitaxy (see figure 2C). Oxide layers 11 and 15 are joined by bonding interface 16.

It is to be specified that the thin film structure serving as seed/joining zone to the bonding interface/substrate may be obtained by other methods than the process described in document FR-A-2 681 472. By way of indication, methods may be cited which are based on bonding by molecular adhesion and thinning by grinding and polishing. It is also possible to use thin layers

transferred by lift-off epitaxy. Numerous examples exist in the literature, in particular to obtain thin films of III-V materials, such as GaAs for example. It is also possible to have recourse to the use of a carrier handle to transfer the thin layers, used as seed, from their basic substrate to the structure which is to become compliant.

To bonding forces it is also possible to make the number of bonding defects (that is to say n ded zones) present on this interface. of the solutions previously put forward, is to ok bonding forces, between the thin film to be used d and the carrier, that are sufficiently low for t in film to absorb stresses without however 15 becom tached. riant of this process consists of exploiting these ng forces and the presence of intermediate layer nce these intermediate layers are able to reinf the compliant nature of the structure. More 20 preci consideration is given in this case, not only e bonding forces between the seed film and the e, but in order to accommodate stresses use is a made of the adhesion forces between the diffe layers and the very nature of the different 25 thin s. e 3 shows, from side view, such compliant subs The compliant substrate 20 comprises a carr , an intermediate layer 22 coated with a thin laye intended to act as seed for hetero-epitaxy. 30 The ediate layer 22 is joined to carrier 21 via a bond terface 24. ay of example, for intermediate layer 22 a meta / be used whose mechanical properties

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(deformation) are such that it may absorb a large part of the stresses. For example, the process described in document FR-A-2 681 472 may be used to obtain the thin film 23 of semiconductor used for germination, but in order to make thin film 23 integral with intermediate layer 22 a metal compound is used containing Au (95%) - Sn (5%) or a compound containing Al (5%) - Cu (95%). These metal compounds have the property of being viscous over a wide temperature range compatible with the temperatures at which epitaxy is generally conducted (900-1000°C). By way of example the use of Pd, Pt may be cited, or of silicides or metal alloys or metal-substrate alloys.

An intermediate layer may also cover the part of the substrate forming the carrier properly so called. This is shown in figure 4 in which the compliant substrate 30 comprises a carrier part 31 coated with a first intermediate layer 32, a second intermediate layer 33 and the thin layer used as seed 34. The bond interface 35 is then situated between the two intermediate layers 32 and 33. These intermediate layers may be of same or different type.

The fabrication of the intermediate layer on the thin film and optionally on the carrier substrate is made before transfer of the intermediate layer/thin film structure used as seed onto the carrier substrate. The intermediate layer is a solid of amorphous, polycrystalline or crystalline type. It may be formed of one or more sub-layers in a same material or a different material and/or be formed of one or more interfaces.

The fabrication of the intermediate layer on the adaptable thin film and optionally on the carrier substrate may be made:

- either using conventional thin layer vacuum
5 depositing techniques (evaporation, cathode spraying, CVD, MBE...),

- or by electrochemical depositing techniques (electrolysis, electroless, etc.),

- or by thin layer transfer techniques: bonding by
10 molecular adhesion then thinning, bonding then thinning using a process such as described in document FR-A-2 681 472, bonding the intermediate layer (already made into a thin layer) via a handle acting as carrier and removal of the handle,

15 - or by conversion of a certain thickness from the surface. This conversion may, for example, be made by oxidation or nitriding. If oxidation is used, it may be either thermal, or anodic, or obtained using another technique (oxygen plasma, oxygen implantation,...).
20 Oxidation may also be conducted through the combination of several oxidation techniques.

-using a method enabling the fabrication of a deformable porous layer.

In the fabrication of a compliant substrate, the
25 thickness of the superficial film may be extremely critical. In some cases, it is necessary to be able to produce superficial films of very narrow thickness. Several methods may be used for the thinning of thin films. In non-exclusive manner the following may be
30 cited: ion abrasion, chemical etching, plasma-assisted etching, laser-assisted ablation, the forming of a sacrificial layer (by oxidation, nitriding the

superficial film...) and removal of this sacrificial layer by various means.

In one application in which the thin layer acting as seed is a silicon film, this film may be the upper
 5 film of a silicon-on-insulator structure, made using the SIMOX technique or a molecular adhesion method, so-called wafer-bonding, for example such as the one described in document FR-A-2 681 472. In this case, the thickness of the silicon film before thinning is for
 10 example, of the order of 0.2 μm . Heat treatment of this superficial silicon film at 1000°C for 70 minutes under a static atmosphere, leads to the formation of a film of silica approximately 0.4 μm thick. On this basis, the superficial silicon film is thinned down
 15 to a low thickness in the order of 1 nm to a few nanometres. Chemical removal of the silica film on the surface is made using 10 % hydrofluoric acid for 10 minutes. This thinning step of the silicon film may usually be completed, for a very thin film of
 20 silica, by heat treatment of the surface under a static atmosphere at high temperature. For example, a treatment at a temperature in the region of 1150°C for 10 minutes enables crystalline reconstruction of the silicon surface. At the same time, thinning of the
 25 silica film of a few nanometres is evidenced. In the approach to compliance, one of the principles is to permit relaxation of epitaxy-related stresses via the film or films of compliance. It may then be advantageous, before epitaxy, to induce a
 30 stress in the superficial film acting as seed, at room temperature, via modification of physical parameters, of mechanical nature, depending upon the type and nature of the depositing to be made. These modifications

are for the purpose of promoting subsequent
rel of deposit stresses. By pre-stressing the
mat it is possible to promote the generation of
dis ons in the superficial film or films of
5 com e or at the interfaces of these films.

eneral, epitaxy is made at a temperature of
sev hundred degrees. The criterion of lattice
ada does not therefore need to be taken into
acc t room temperature. It is important to assess
10 the of the stresses of thermal origin related, for
exa to differences in thermal dilatation between
the ous films and the mechanical carrier
(sul e).

his optic, the fact may be used that it is
15 pos to modify the crystalline parameter of the
sup al film using implantation by bombardment of
an e in the crystalline matrix of the superficial
film ionally supplemented by heat diffusion of the
eler One variant of implantation by bombardment is
20 to e processes based solely on thermal diffusion
of ts, such as the diffusion of doping agent in
sili By way of example of ion implantation, mention
may ade of boron implantation in monocrystalline
sili This leads to a reduction in crystalline
25 latt e 0.014 Å/atom % of the species inserted. If
the ficial film adheres strongly to the mechanical
carr the silicon film will then be placed in a
tens ate. In the same way, the effect of germanium
impl on will be to increase the crystalline
30 latt y 0.0022 Å /atom %. If the superficial film
adhe rongly to the mechanical carrier, the film of
sili ll then be placed in a state of compression.

the case of thin silicon films, made for
 co nce by thinning through sacrificial oxidation as
 de ed previously, implantation may advantageously
 be before removal of the oxide. The film of oxide
 5 th re acts as protective film during heat diffusion
 t nt of the implanted element. By way of example
 i ation of boron at an energy in the region of 110
 k th a dose in the order of a few $10^{15}/\text{cm}^2$ to a few
 10 via the oxide layer with a thickness close to
 10 0 leads to enriching the very thin silicon film
 w oron by coinciding the depth of this film with
 t th of ion implantation. Stresses of a few 10^8MPa
 m ce be generated in the thin film of silicon.

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CLAIMS

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1. Compliant substrate (5, 20, 30) comprising a carrier (1, 14, 21, 31) and at least one thin layer (4, 13, 23, 34) formed on the surface of said carrier and
5 intended to receive, in integral manner, a stress-giving structure, the carrier and the thin layer being joined one to another by joining means (3; 11, 15, 16; 24, 25) such that the stresses brought by said structure are absorbed in whole or in part by the thin
10 layer and/or the joining means, characterized in that said joining means comprise at least one joining zone chosen from among the following joining zones: a layer of microcavities and/or a bonding interface whose bonding energy is controlled to permit the absorption
15 of said stresses.

2. Process for fabricating a compliant substrate according to claim 1, characterized in that the layer of microcavities is created through implantation by
20 bombardment of one or more gas species.

3. Process according to claim 2, characterized in that the gas species are chosen from among rare gases, hydrogen and fluorine.
25

4. Process according to either of claims 2 or 3, characterized in that doping agents are associated with the one or more gas species.

30 5. Process according to claim 2, characterized in that diffusion is made of the one or more implanted gas species.

6. Process according to claim 2, characterized in that implantation is followed by heat treatment.

5 7. Process according to any of claims 2 to 6, characterized in that said implantation is made via the substrate surface, the region lying between the substrate surface and the layer of microcavities providing said thin layer.

10

8. Process according to claim 7, characterized in that the region lying between the substrate surface and the layer of microcavities is thinned to form said thin layer.

15

9. Process according to claim 7, characterized in that implantation by bombardment is made via a sacrificial layer (2) carried by said substrate surface, said sacrificial layer then being removed.

20

10. Process according to any of claims 2 to 6, characterized in that said implantation is made via the substrate surface, this surface carrying a first thin layer, the region lying between the substrate and the layer of microcavities providing a second thin layer.

25

11. Process according to claim 10, characterized in that the layer of microcavities is made in the vicinity of the interface between the first thin layer and the substrate.

30

12. Process according to either of claims 10 or 11, characterized in that implantation by bombardment

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is made via a sacrificial layer carried by the first thin layer, said sacrificial layer then being removed.

13. Compliant substrate according to claim 1,
5 characterized in that bonding energy is controlled by surface preparation and/or heat treatment and/or creation of defects at this interface.

14. Compliant substrate according to claim 13,
10 characterized in that surface preparation is a control of roughness and/or hydrophilia.

15. Compliant substrate according to either of claims 13 or 14, characterized in that said joining
15 zone also comprises at least one intermediate layer (22; 32, 33) between the thin layer (23; 34) and the carrier (21; 31).

16. Compliant substrate according to claim 15,
20 characterized in that the intermediate layer (22; 32, 33) is a metal layer or metal alloy layer.

17. Compliant substrate according to claim 15,
25 characterized in that at least one intermediate layer is formed such that it is made up of non-homogeneities able to relax stresses.

18. Compliant substrate according to any of claims 1 to 17, characterized in that the joining means
30 comprise a layer of microcavities and a bonding interface arranged either above or below the layer of microcavities.

19. Compliant substrate (5, 20, 30) according to any of the preceding claims, characterized in that said thin layer (4, 13, 23, 34) is in a first crystalline material and is intended to be used as hetero-epitaxial growth seed for a second crystalline material forming said structure.

20. Compliant substrate according to claim 19, characterized in that said thin layer is a pre-stressed layer through the insertion of a foreign element in said first crystalline material in order to promote the compliance of said substrate.

21. Compliant substrate according to claim 20, characterized in that the foreign element is inserted through implantation by bombardment and/or inserted by diffusion.

22. Compliant substrate according to either of claims 20 or 21, characterized in that said foreign element is a doping agent of the thin layer.

23. Compliant substrate (5, 20, 30) according to any of claims 19 to 22, characterised in that said first crystalline material is a semiconductor.

24. Application of the compliant substrate (5, 20, 30) according to any of claims 19 to 23, to the hetero-epitaxial growth of a crystalline material chosen from among GaN, SiGe, AlN, InN, and SiC.

ABSTRACT OF THE DISCLOSURE

COMPLIANT SUBSTRATE

IN PARTICULAR FOR HETERO-EPITAXIAL DEPOSITING

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The invention relates to a compliant substrate (5) comprising a carrier (1) and at least one thin layer (4), formed on the surface of the carrier and intended to receive, in integral manner, a stress-giving structure. The carrier (1) and the thin layer (4) are joined to one another by joining means (3) such that the stresses brought by said structure are absorbed in whole or in part by the thin layer (4) and/or by the joining means (3) which comprise at least one joining zone chosen from among the following joining zones: a layer of microcavities and/or a bonding interface whose bonding energy is controlled to permit absorption of said stresses.

15 Figure 1C

1/4

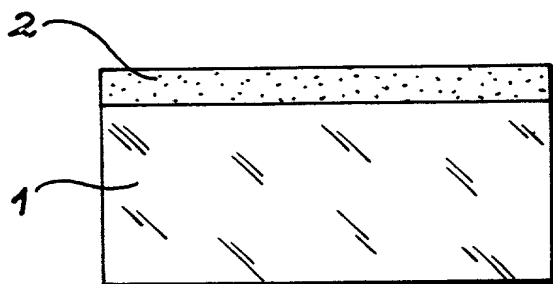


FIG. 1A

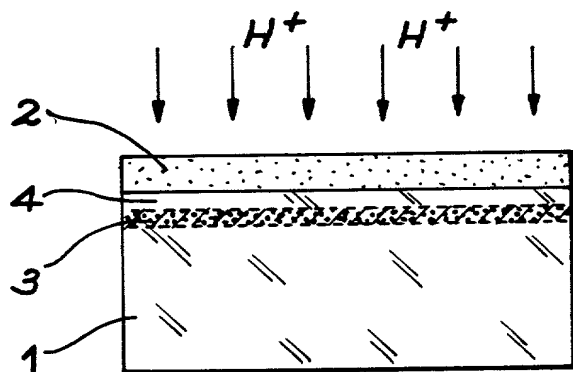


FIG. 1B

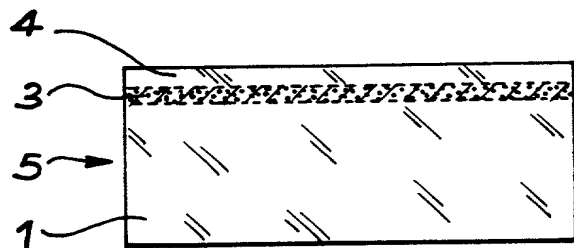


FIG. 1C

2/4

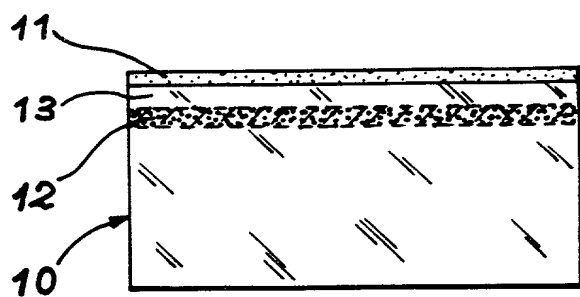


FIG. 2A

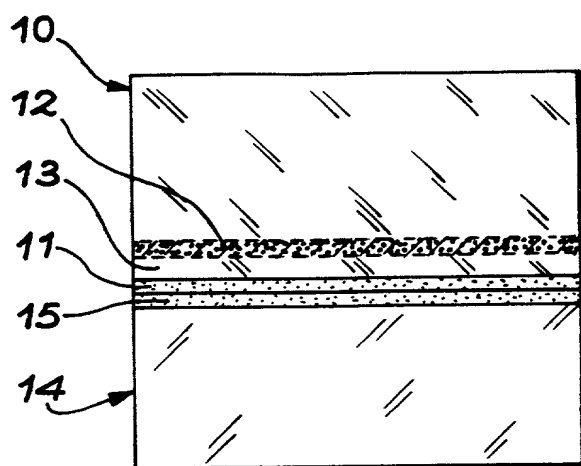


FIG. 2B

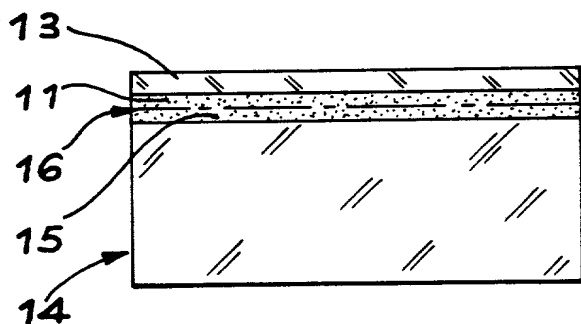


FIG. 2C

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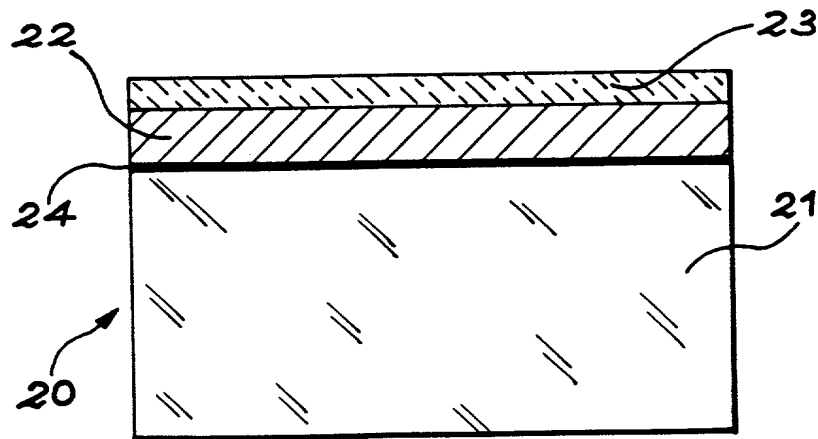


FIG. 3

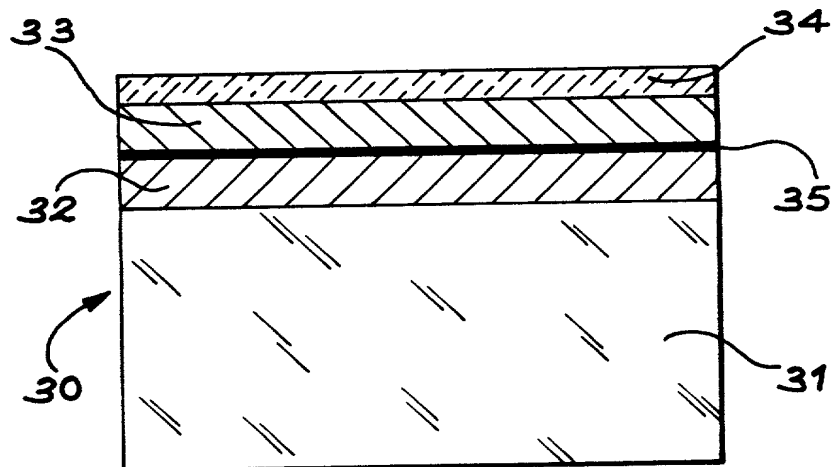


FIG. 4

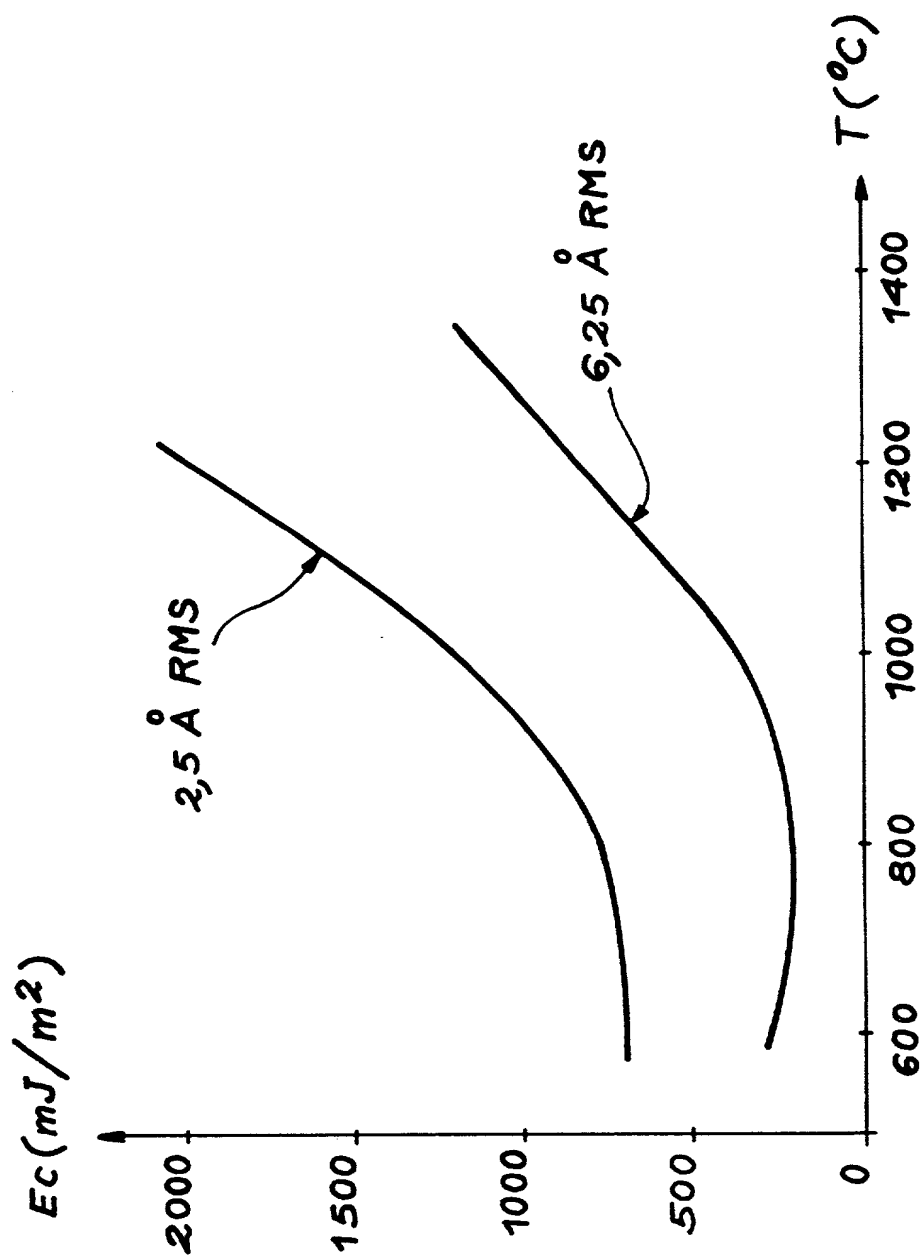


FIG. 5

Declaration, Power Of Attorney and Petition

Page 1 of 3

WE (I) the undersigned inventor(s), hereby declare(s) that :

My residence, post office address and citizenship are as stated below next to my name,

We (I) believe that we are (I am) the original, first, and joint (sole) inventor(s) of the subject matter which is claimed and for which a patent is sought on the invention entitled

"Compliant substrate in particular for hetero-epitaxial depositing"

the specification of which

- ☐ is attached hereto.
- ☐ was filed on
as Application Serial No.
and amended on
- ☒ was filed as PCT international application
Number PCT/FR99/00187
on January 29, 1999
and was amended under PCT Article 19
on February 21, 2000

We (I) hereby state that we (I) have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

We (I) acknowledge the duty to disclose information known to be material to the patentability of this application as defined in Section 1.56 of Title 37 Code of Federal Regulations.

We (I) hereby claim foreign priority benefits under 35 U.S.C. § 119 (a)-(d) or § 365 (b) of any foreign application(s) for patent or inventor's certificate, or § 365 (a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed. Prior Foreign Application (s)

Application No.	Country	Day/month/Year	Priority Claimed	
98 01061	FRANCE	30 JANUARY 1998	<input checked="" type="checkbox"/> YES	<input type="checkbox"/> NO
_____	_____	_____	<input type="checkbox"/> YES	<input type="checkbox"/> NO
_____	_____	_____	<input type="checkbox"/> YES	<input type="checkbox"/> NO
_____	_____	_____	<input type="checkbox"/> YES	<input type="checkbox"/> NO

We (I) hereby claim the benefit under Title 35, United States Code, § 119 (e) of any United States provisional application(s) listed below.

(Application Number)

(Filing Date)

(Application Number)

(Filing Date)

We (I) hereby claim the benefit under 35 U.S.C. §120 of any United States application(s), or § 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56 which became available between the filing date of prior application and the national or PCT International filing date of this application.

Status (pending, patented,

Application Serial No.

Filing Date

abandoned)

And we (I) hereby appoint :William L. Mathis, Registration Number 17,337; Alan E. Kopecki, Registration Number 25,813; Eric H. Weisblatt, Registration Number 30,505; Peter H. Smolka, Registration Number 15,913; Regis E. Slutter, Registration Number 26,999; James W. Peterson, Registration Number 26,057; Robert S. Swecker, Registration Number 19,885; Samuel C. Miller III, Registration Number 27,360; Teras Stanek REA, Registration Number 30,427; Platon N. Mandros, Registration Number 22,124; Ralph L. Freeland Jr., Registration Number 16,110; Robert E. Krebs, Registration Number 25,885; Benton S. Duffett jr., Registration Number 22,030; Robert M. Schulman, Registration Number 31,196; Joel M. Freed, Registration Number 25,101; James A. Labarre, Registration Number 28,632; William C. Rowland, Registration Number 30,888; Norman H. Stepno, Registration Number 22,716; E. Joseph Gess, Registration Number 28,510; Richard H. Kjeldgaard, Registration Number 30,186; Ronald L. Grudziecki, Registration Number 24,970; David D. Reynolds, Registration Number 29,273; T. Gene Dillahunt, Registration Number 25,423; Frederick G. Michaud Jr, Registration Number 26,003; R. Danny Huntington, Registration Number 27,903 and Anthony W. Shaw, Registration Number 30,104; our (my) attorneys, with full powers of substitution and revocation, to prosecute this application and to transact all business in the Patent Office connected therewith; and we (I) hereby request that all correspondence regarding this application be sent to the firm of BURNS, DOANE, SWECKER & MATHIS, whose post Office Address is : George Mason Building, Washington and Prince Streets, P.O. Box 1404 Alexandria, Virginia 22313-1404.

We (I) declare that all statements made herein of our (my) own knowledge are true and that all statements made on information and belief are believed to be true ; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such wilful false statements may jeopardise the validity of the application or any patent issuing thereon.

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Signature of Inventor

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